

FOURTH QUARTERLY REPORT  
TO  
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
GODDARD SPACE FLIGHT CENTER  
COVERING

t: ALKALINE BATTERY SEPARATOR STUDY

March 28, 1963 - June 28, 1963

Contract NASX5-2860 )

The Carl F. Norberg Research Center  
The Electric Storage Battery Company  
Yardley, Pennsylvania  
Pa.

July 11, 1963

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## I. Status Summary

### A. Introduction

This is the fourth quarterly report covering work during the reporting period March 28, 1963 through June 28, 1963 on Contract NAS-5-2860, which was awarded to the Electric Storage Battery Company by the National Aeronautics and Space Administration, Goddard Space Flight Center, on June 28, 1962.

### B. Previous Results

Degradation studies on cellophane showed that oxidation, rather than hydrolysis, determined the decay of physical properties. The permanganate method gave the best reproducibility. Data on the dimensional stability in electrolyte at various temperatures, electrical resistivity, absorption and retention of electrolyte were obtained for various separator materials.

## II. Work Accomplished During the Reporting Period

### A. Cellophane Degradation

The alkaline hydrolysis of cellophane in the presence and absence of air has been discussed in previous reports under this contract. In the absence of air, the degree of polymerization of cellophane did not decrease appreciably after 72 hours. In the presence of air, an irregular trend occurred in the degree of polymerization caused by limited oxygen availability. However, by bubbling gaseous oxygen through the 40% alkaline solution containing cellophane, the degree of polymerization showed a regular, rapid downward trend.

In the literature<sup>1</sup>, oxidation of cellulose by oxygen in an alkaline medium is reported to proceed by a hydroperoxide-type, free radical mechanism, catalyzed by metals such as iron, cobalt and manganese. On the other hand, silver is reported to inhibit the oxidation of cellophane. In the light of this, experiments have been initiated to determine the effect of zincate on the rate of cellulose degradation.

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To measure the hydrolytic effect, samples of cellophane, 8 1/2 x 4 in., were suspended on a Teflon frame and immersed in a resin kettle filled with 31% KOH which was saturated with ZnO. The kettle, fitted with a condenser, drying tube, and a nitrogen bubbler was placed in a constant temperature bath at 50°C; samples were removed at specified time periods, solution viscosities obtained and DP values calculated. Under these conditions there was no appreciable change in degree of polymerization after 80 hours. Samples of cellophane for tensile strength were prepared in the same manner; tensile values exhibited an initial increase in strength but no further change. Table I shows the changes in degree of polymerization and in breaking load.

TABLE I

Cellophane Hydrolysis with Gaseous Nitrogen  
 in 31% KOH - ZnO Saturated

Time in Hours	Degree of Polymerization	Breaking Load (lbs.)
0	520	13
8	505	16
24	487	16
32	478	—
48	474	17
56	463	—
72	453	16
80	453	16

The experiments were repeated under oxidizing conditions by replacing the nitrogen with oxygen. A marked decrease in the degree of polymerization occurred in the first 24 hours. After 48 hours the samples began to disintegrate. Tensile strength measurements were made but after 48 hours, the samples were sufficiently degraded to make tensile measurements impractical. Table II shows the changes in degree of polymerization and breaking load. Figure I shows the decreases in the degree of polymerization of cellophane in zinc saturated electrolyte under nitrogen and under oxygen.

TABLE II

Cellophane Hydrolysis with Gaseous Oxygen  
 in 31% KOH - ZnO Saturated

Time in Hours	Degree of Polymerization	Breaking Load (lbs.)
0	520	13
8	461	13
24	342	8
48	262	—
56	235	—
72	214	—

Additional experiments are under way to examine the concentration effects of both zincate and alkali.

B. Oxidation Test

Oxidation resistance of separator materials was studied in the presence of sodium hypochlorite, gaseous oxygen and potassium permanganate. The potassium permanganate method was found to be the most reproducible and applicable to all separator materials under study.

Samples 3 x 2 in. were dried to constant weight and then immersed in a solution of 250 ml 5%  $\text{KMnO}_4$  and 50 ml 25%  $\text{NaOH}$  for 1 hour at  $50^\circ\text{C}$ . (The procedure is given in detail in the previous report, ESB Report No. 7054.3) Since the cellophane samples were virtually destroyed under these conditions, the temperature was reduced to  $25^\circ\text{C}$  for cellulosic films. The sample was again dried to constant weight and from the difference in weight between the original and the final weight, percentage loss was recorded. The following table summarizes the results of the potassium permanganate oxidation test for the separator materials tested to date.

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TABLE III

Oxidative Resistance in  $\text{KMnO}_4$  at  $50^\circ\text{C}$  for 1 hr.

Material	Percent wt loss
Cellophane PUDO-300*	69
Cellophane PUDO-600*	63
Cellophane 140-PUT-76*	92
Cellophane Gel Film*	95
Fibr. Saus. Casing	38
Silvered Saus. Casing	31
Permion 300	23
Permion 600	41
Polypor WA	7
Mipor 12CN	1.2
Mipor 13CN	0.8
Mipor 34CN	1.5
Mipor 34PN	0.9
Cellophane 111-1*	32
Cellophane 111-2*	60
Acropor WA	0

\* Test run at  $25^\circ\text{C}$  for 1 hour

### C. Cellophane Modification

Because of its polyhydric alcohol structure, cellulose is extremely sensitive to oxidation, in the presence of alkali. This oxidation tendency should be inhibited by blocking the sensitive groups, rendering them less susceptible to attack.

All cellophanes are partially oxidized and contain therefore both carboxyl and aldehyde groups. Cations can be bound to cellulose through their weakly acid functional groups. After the cations are bound within the cellulose film, redox type polymerization can be initiated at the site of the bound cations.

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Grafting of olefin polymers onto cellulose molecules by utilizing the ion exchange capacity of the cellophane functional groups<sup>2</sup> was initiated in an attempt to increase the resistance of cellophane to oxidation.

Previously weighed cellophane strips were immersed in a .1%  $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4$  solution for 5 minutes and then washed to remove the uncombined iron. They were then placed in a 1%  $\text{H}_2\text{O}_2$  solution for 1 second. The films were immersed in refluxing 5% aqueous acrylonitrile solution for varying lengths of time. Upon removal from the solution, the films were extracted with dimethyl formamide to remove non-grafted polyacrylonitrile and the weight increase measured. Oxidation resistance and electrical resistance were also measured. The results are given in Table IV.

In other experiments, methacrylic acid monomer was used. The procedure differed in that the hydrogen peroxide was added to the refluxing monomer solution before the conditioned film was dropped into the solution. Cellophane films were steeped in a .1%  $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4$  solution and washed with distilled water. They were dropped into 200 ml .003%  $\text{H}_2\text{O}_2$  solution containing 10 ml methacrylic acid. The films were removed after specified time periods and extracted with methyl ethyl ketone to remove any non-grafted polymethacrylic acid. Weight increase was measured as well as oxidation and electrical resistance. These results are also shown in Table IV.

TABLE IV

Material	Percent wt. increase	Percent wt. Loss in $\text{KMnO}_4$ at 25°C for 1 hr.	Resistance OHMS for sq. in.
Cellophane 300-PUDO	0	69	.0098
Cellophane 300-PUDO- Polyacrylonitrile	85	41	.0833
Cellophane 300-PUDO Polymethacrylic Acid	10	68	.0196



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The films treated with methacrylic acid showed no substantial weight increase. Polymerization of the methacrylic acid monomer solution occurred very rapidly and could not be limited to the grafting sites. The cellophane films had to be removed before they became excessively coated with polymethacrylic acid, causing embrittlement of the films after extraction. Contact time could not be controlled sufficiently to give intermediate values of grafting thus accounting for the small increase in weight of the films.

In these preliminary experiments, polymerization was not confined to the grafting site but homopolymerization also occurred, resulting in the coating of the sample at longer reaction times. Experimental conditions will be modified to permit adequate control of the reaction.

#### D. Diffusion Rate of Silver Ions

Experimental work was initiated to study the diffusion rate of silver ions through the various separator materials. The method consists in filling both compartments of a diffusion cell with 31% KOH, adding to one compartment silver ions in form of  $\text{AgNO}_3$  solution, tagged with 1 micro-curie  $\text{Ag}^{110}$ , (obtained from the Oak Ridge National Laboratory) then determine the rate at which the radioactivity diffused into the other compartment through the various separator materials.

The cell is shown in Figure 2 and in Photographs No. 1 and 2. It consists of two compartments, machined from cast poly (methyl methacrylate) rod. A specimen, 1.5 in. in diameter, is cut with a steel die from the membrane to be tested. The specimen is clamped, together with a rubber gasket, between the two compartments of the cell. The gasket seal around the edge of the membrane was found to be leakproof in a test in which non-porous polyethylene film was substituted for the separator sample: in 114 hours, less than 0.1% of the radioactive silver originally present had leaked through the seal.

The diffusion rate was determined by withdrawing, at suitable intervals from zero time, 1 ml. samples of the solution from each compartment and measuring the radioactivity of each sample, using a gamma scintillation counter (Atomic Instruments Co., Cambridge, Mass.)

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Experimentally, there were two problems that appeared when this method was used in testing cellophane.

It was found that a large fraction of the radioactive silver was deposited on the walls of the cell, making it unavailable for diffusion. Different methods of preparing the tracer solution will be studied.

Another problem is the chemical reaction which appears to occur between the dissolved silver and the cellophane. The silver oxidizes the cellulose and is reduced to metal. It deposits in the cellophane at such a rate that very little radioactive silver remains in solution after one to two days while scarcely any is detectable in the initially non-radioactive compartment. Thus, membrane reactions rather than diffusion takes place. This observation confirms Prof. Dirkse's findings<sup>3</sup>, namely, that the cellophane traps the silver ions and does so with almost 100% efficiency. The data listed in Table V illustrates the problem.

TABLE Va

Diffusion through Cellophane

Time (hours)	Compartment I	Compartment II	% of total
0-15	20690	14 ± 6	= 0.07
21-30	9000	28 ± 7	= 0.30
31-40	2050	23 ± 6	= 1.10

TABLE Vb

Diffusion of Silver through Cellophane

Time (hours)	Compartment I	Compartment II	% of total
0-15	20690	14 ± 6	= 0.07
21-30	9000	28 ± 7	= 0.30
31-40	2050	23 ± 6	= 1.10

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There is thus very little difference in the diffusion rate between the non-porous polyethylene blank and cellophane. This is in marked contrast with a non-cellulosic material, as exemplified by Acropor WA.

TABLE Vc

Diffusion through Acropor WA

Time (hours)	Compartment I	Compartment II	% of total
0.5	11050	72 ± 7	0.65
5.5	9645	340 ± 10	3.40
18.5	7640	540 ± 13	6.60

These figures show that, in the case of Acropor WA, appreciable diffusion is taking place.

Future work will attempt to solve these problems so that diffusion can be studied with this method. The study will be extended to other, non-cellulosic materials.

E. Measurement of Average Pore Size of Separator Materials

The established methods for the measurement of pore size distribution in separator materials, such as infusion with mercury under pressure, are in general limited to dry materials. Since most of the materials used in alkaline batteries swell a great deal in the electrolyte, dry measurements of their pore size distribution may not be significant. However, methods exist to measure an average effective pore size and a maximum pore size in battery electrolyte.

The method selected for the calculation of the average pore size is that of Bruins, Gregor, Bieber, Capsuto and Salkind<sup>4</sup> which has also previously been reported by the Missile Battery Division of ESB<sup>5</sup>. In this method, based on Poiseuille's law, the time for a volume of electrolyte to flow through the separator from a given height is determined and, from this data and other measured properties of the separator, the average pore size is calculated.

The apparatus used in our measurements is shown in Figure 3. It consists of a .070"ID (.178 cm) riser tube, a special polyethylene separator holder which exposes a 1 cm diameter opening for the electrolyte flow and a meter stick assembly. An essential precaution in using the apparatus is that the change in the liquid level in the beaker be very small during flow.

The equation applied was:

$$[1] \quad r^2 = -\left[\ln \frac{h}{H_1}\right] \frac{a}{AB} \left[\frac{8\mu L^2}{9\rho}\right] \frac{1}{t}$$

These parameters have the following meaning:

$r$  = radius of pore.

$H$  = original height of electrolyte in riser tube above separator, at start of timed flow period.

$h$  = final height.

$a$  = Cross sectional area of riser tube.

$A$  = Exposed separator cross sectional area.

$B$  = Total pore volume of separator/per unit surface.

$\mu$  = Viscosity of electrolyte.

$\rho$  = Density of electrolyte.

$g$  = Gravitational constant, 980 cm/sec<sup>2</sup>

$t$  = Fall time in seconds.

The parameter which is most difficult to measure precisely is  $B$ , the total pore volume/unit surface. Commonly, this has been measured in the past<sup>4</sup> by soaking separators in water and measuring the gain after blotting the

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separator free of excess water. It has been shown previously by Chreitzberg<sup>5</sup> and confirmed in a series of experiments discussed a little later in this report, that a more accurate measure of the total porosity is obtained by soaking the separator materials in electrolyte and, after allowing them to air drip for 1 minute, blotting the bottom edge only before weighing.

For the particular apparatus used and with 31% KOH as the electrolyte, equation [1] can be simplified as follows:

$$[2a] \quad \frac{a}{A} \frac{8}{g_p} \frac{\mu}{.785 \text{ cm}^2} = \frac{.025 \text{ cm}^2}{(980 \text{ cm/sec}^2)} \frac{(8) (.022 \text{ gm/cm-sec})}{1.30 \frac{\text{gm}}{\text{cm}^3}}$$

$$= 4.4 \times 10^{-6} \text{ cm-sec}$$

and

$$[2b] \quad r^2 = 2.3 \log \frac{H}{h} \frac{L^2}{Bt} [4.4 \times 10^{-6} \text{ cm-sec}]$$

when the parameters in [2b] are in the following units:

$h, H$  in cm,

$L$  = thickness of sep. in cm,

$B$  = pore volume/unit surface, in  $\text{cm}^3/\text{cm}^2$ ,

$t$  = time, seconds; then

$r$  will be in cm.

As previously mentioned, one of the uncertainties in the use of equation [1] is the precision with which the value  $B$  can be determined. To evaluate procedures, we made use of two instruments which determine total porosity on dry separators. These were the Beckman Air Pycnometer and the Aminco-Winslow Porosimeter. We measured the porosity of dry Synpor, (an ESB proprietary porous polyvinyl chloride membrane), which does not swell on

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immersion in electrolyte, and then compared this value with the values obtained by wet methods. This was also done with Acropor WA (Gelman Ind.), a material which does swell slightly in electrolyte. In comparing these values, one cannot expect perfect agreement. All pores are not through pores with no tortuosity, as is assumed in equation [1]. Also, the mercury porosimeter may distend the pores while measuring them, at the relatively high pressure involved (5000 psi). In using the Beckman Air Pycnometer to calculate porosity, the following equation was applied:

$$[3] \text{ Porosity} = \frac{\text{Apparent Volume Sep.} - \text{True Volume}}{\text{App. Vol.}}$$

With the mercury porosimeter, the following equation was applied:

$$[4] \text{ Porosity} = \frac{\text{Pore/unit wt} = \text{cc/gm}}{\text{App. vol. cc/gm}}$$

The values obtained show good experimental correlation as shown in Table VI.

TABLE VI

Values of Separator Porosity

<u>ρ App</u> gm/cc	<u>ρ True</u> gm/cc	<u>Pore Vol (Hg)</u> cc/gm	<u>Porosity cc</u> <u>Pore cc Super-</u> <u>ficial Vol.</u>	<u>"B" cc</u> <u>Pore cm<sup>2</sup></u> <u>Surface</u>	<u>Method</u>
A) Synpor (.02 cm thick)					
1) .277	—	2.8	.77	.0157	mercury porosimeter
2)	Water Absorption Method			.0166	bottom blot
3)	Water absorption Method			.0163	surface blot
4)	Water Absorption Method			.0155	air dry 1 min bottom blot
5) .277	1.5	—	.81	.0162	(equation 3)

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(TABLE VI Cont.)

$\rho_{App}$ gm/cc	$\rho_{True}$ gm/cc	Pore Vol (Gh) cc/gm	Porosity cc Pore cc Super- ficial Vol.	"B" cc Pore cm <sup>2</sup> Surface	Method
B) Acropor WA (.01 cm thick)					
1) .526	1.41	—	.61	.0061	equation 3
2) —	—	—	.20	.0020	water absorption
3) —	—	—	.50	.0050	electrolyte absorption
4) .526	—	1.03	.54	.0054	mercury porosimeter

The pore size distributions for Synpor and Acropor WA were taken on the dry material by mercury porosimeter. The results are shown in Figures 4 and 5. The values obtained for the electrolyte flow through Acropor WA are plotted in Figure 6 as log H/h vs. time. The straight line indicates the data were obtained (as needed) in the viscous flow region. The calculations for the average pore size are given in Table VII. Equation 2b was used to calculate "r".

TABLE VII

Calculations for Average Pore Size

<u>Material</u>	<u>Synpor</u>	<u>Acropor WA</u>
L	.02	.01
B	.0157	.005
log $\frac{H}{h}$	.301	.175
t sec	7.2	15.5
$r^2$	$1.9 \times 10^{-8}$	$23 \times 10^{-10} \text{ cm}^2$
d	70,000	9,600
pore diam:	Approx. 7 $\mu$	Approx. 1 $\mu$

These values compare favorably with the sizes for pores shown in figures 3 and 4.

Data are now being obtained with the other separator materials under test. Values for maximum pore size will also be calculated.

#### F. Dimensional Change Measurements

Tables showing the dimensional changes on prolonged immersion in electrolyte were attached to the previous report (3rd Quarterly Report, ESB No. 3, Tables 8 through 10). The data were incomplete at the time. Additional data on dimensional changes have been secured and are contained in Tables 8, 9 and 10 of the current report. For ease of comparison, these tables repeat the pertinent data for cellophane PUDO-300 (for comparison with the same cellophane, impregnated with m-phenylene diamine) and for Permion 300 (for comparison with other polyethylene based materials, the PMA's).

#### G. Summary of the Work to Date

The bench tests which had been selected have proved to be valuable tools for screening separator materials. In the aggregate, they permit conclusions to be drawn regarding the probable suitability of any specific material.

Twenty-six different separator materials were compared. Some of these were commercial products, others were of an experimental nature. The experimental data covering electrical resistance, electrolyte absorption and retention, tensile strength, dimensional stability and oxidation resistance in the battery environment, lead to the following conclusions:

1. The celluloseics have the lowest electrical resistivity of all the materials considered, coupled with the highest values for electrolyte absorption and retention. However, their oxidation resistance is very poor and they undergo considerable thickness swelling in the electrolyte.
2. The silvered sausage casing demonstrates that the low electrical resistivity of the celluloseics is further enhanced by treatment of celluloseics with a silver salt solution and subsequent reduction of the silver ion to metallic silver. Electrolyte absorption and retention are thereby reduced but remain satisfactory. The oxidation resistance and tensile strength are also markedly improved though the oxidation loss still remains fairly high.



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3. One of the experimental cellophanes (DuPont No. 111-1) shows some improvement when the cellulose was linked with epoxy compounds. Both oxidation loss and swelling in electrolyte are reduced while the electrical resistivity is only slightly increased over conventional cellophane; the electrolyte absorption and retention values are lower, as expected, but the reduction is not too serious. Cross-linking appears thus to hold some promise as a means for up-grading of cellophane. This approach is therefore being pursued further.
4. Permion 600, which is described as a modified cellulosic material, has a somewhat better oxidation resistance but the resistivity is appreciably higher and the absorption and retention values are lower than in unmodified cellulose separators.
5. Of the non-cellulosic materials, Acropor WA comes closest to cellophane in its low electrical resistivity. Its oxidation resistance is considerably higher and its dimensional stability in electrolyte is also markedly better. However, its electrolyte absorption and retention is sharply reduced, compared with cellophane.
6. The PMA membranes which are made by polymerizing and cross-linking methacrylic acid on a microporous polyethylene base also couple good oxidation resistance with reasonable electrical resistivity. Both properties are dependent on the degree of cross-linking.
7. One of the Mipor samples, i.e. Mipor 13 CN, a microporous polyethylene of high porosity, is even better in oxidation resistance, in fact, its oxidation loss is negligible. This is coupled with an electrical resistivity which is only very little lower than that of Acropor WA. However, while its electrolyte absorption and retention values are better than those of Acropor WA, they are still very much lower than the corresponding values for cellophane.
8. Permion 300 which has also a polyethylene basis has good electrolyte absorption and retention characteristics. Its oxidation resistance, while appreciably better than that of cellulose, is markedly lower than that of other polyethylene-based materials.

9. The AMFion membranes have such high electrical resistivities that they cannot be considered as suitable separator materials.

#### Future Work

Based on the information derived from this test program, two approaches to improved separator materials have emerged:

- a. Since cellulosic materials have proved satisfactory in most other respects, ways must be sought so to modify the cellulose molecule as to improve drastically its chemical stability, particularly its resistance to oxidative degradation.
- b. A second approach must start with those materials which have proved highly resistant to chemical degradation but which are deficient either in electrical resistivity or in their ability to absorb and retain the electrolyte. Either chemical or physical modifications, or combinations of both, are indicated.

Work along both these lines is being actively pursued. Various monomers have been graft polymerized onto the cellulose molecule; means of cross-linking are also being explored. Ways to modify oxidation resistant polymers so as to improve their electrical resistance are being investigated. All new or modified materials are and will be evaluated by the methods which have been described above.

#### Bibliography

1. Ott, Emil, Cellulose and Cellulose Derivatives, New York, Interscience Publishers, 1954.
2. Bridgeford, D. J., "Catalytic Deposition and Grafting of Olefin Polymers onto Cellulosic Materials", Ind. and Eng. Chem., R. and D., 1, 45-51 (1962).
3. Dirkse, T. P., van der Lugt L. A. - Second Quarterly Report, Contract AF 33 (657)-8689; Nov. 1, 1962.
4. Bruins, P. F., Gregor, H. P., Bieber, H. H., Capsuto, L. and Salkind, A. J., Polytechnic Institute of Brooklyn, Chemical Engineering Department, Contract DA-36-039 SC 70118 May 31, 1956, page 14.
5. Final Report, NASA, Goddard Space Flight Center, Contract NAS-5-1607, ESB Report No. E-6-63.

TABLE VIII

% Dimensional Changes in 31% KOH at -26°C

	70 hours		7 days		14 days	
	Length	Width	Length	Width	Length	Width
1 Cellophane PUDO-300	-5	+10	+300	-5	+10	+300
25 Cellophane PUDO-300, impreg.	-2	+25	+170	-2	+25	+170
11 Permion 600	0	+5	+35	0	+5	+10
15 PMA/Polyethylene, high crosslink	+10	+10	-10	+10	+10	-25
17 PMA/Polyethylene, low crosslink	+10	+20	+45	+10	+20	+35
26 Acropor WA	0	0	0	0	0	-5

	21 days		28 days	
	Length	Width	Length	Width
1	-10	+10	+280	-10
25	-3	+25	+140	-2
11	0	0	0	0
15	+10	+10	-25	+10
17	+10	+20	+40	+20
26	0	0	-5	0

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TABLE IX

% Dimensional Changes in 31% KOH at 26°C

	70 hours			7 days			14 days		
	Length	Width	Thickness	Length	Width	Thickness	Length	Width	Thickness
1 Cellophane PUDO-300	-5	+5	+280	-5	+5	+200	+5	+5	+170
25 Cellophane PUDO-300, impreg.	0	+10	+70	0	+10	+80	0	+10	+75
11 Permion 600	+5	+10	+25	+5	+5	0	+5	+10	+10
15 PMA/Polyethylene, high crosslink	+8	+12	0	+7	+10	+30	+7	+10	+5
17 PMA/Polyethylene, low crosslink	+15	+30	+30	+15	+30	+30	+15	+30	+30
26 Acropor WA	0	0	+5	0	0	+10	0	0	+10

	21 days			28 days		
	Length	Width	Thickness	Length	Width	Thickness
1	*	*	*	*	*	*
25	0	+10	+60	0	+10	+80
11	+5	+10	+10	+5	+10	+50
15	+8	+10	-3	+8	+10	-3
17	+15	+30	+30	+15	+30	+30
26	0	0	+10	0	0	+10

\* Tests discontinued due to sample deterioration.

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TABLE X

% Dimensional Changes in 31% KOH at +90°C

	70 hours			7 days			14 days		
	Length	Width	Thickness	Length	Width	Thickness	Length	Width	Thickness
1 Cellophane PUDO-300	-25	+15	+280	-25	+10	+300	-25	+15	+310
25 Cellophane PUDO-300, impreg.	0	+10	+70	0	+10	+65	0	+10	+45
11 Permion 600	-5	+10	+45	-10	+5	+50	-10	0	+36
15 PMA/Polyethylene, high crosslink	+7	+11	+30	+8	+11	+33	+8	+13	+30
17 PMA/Polyethylene, low crosslink	+20	+34	+40	+20	+35	+40	+20	+35	+35
26 Acropor WA	0	0	0	0	0	+10	-2	0	-5

21 days		
Length	Width	Thickness
-30	+5	+260
0	+10	+80
-10	-5	+22
+7	+10	+30
+20	+35	+50
-2	0	-2

Cellophane Hydrolysis at 50°C in 31% KOH - ZnO Saturated  
Under Nitrogen and Under Oxygen

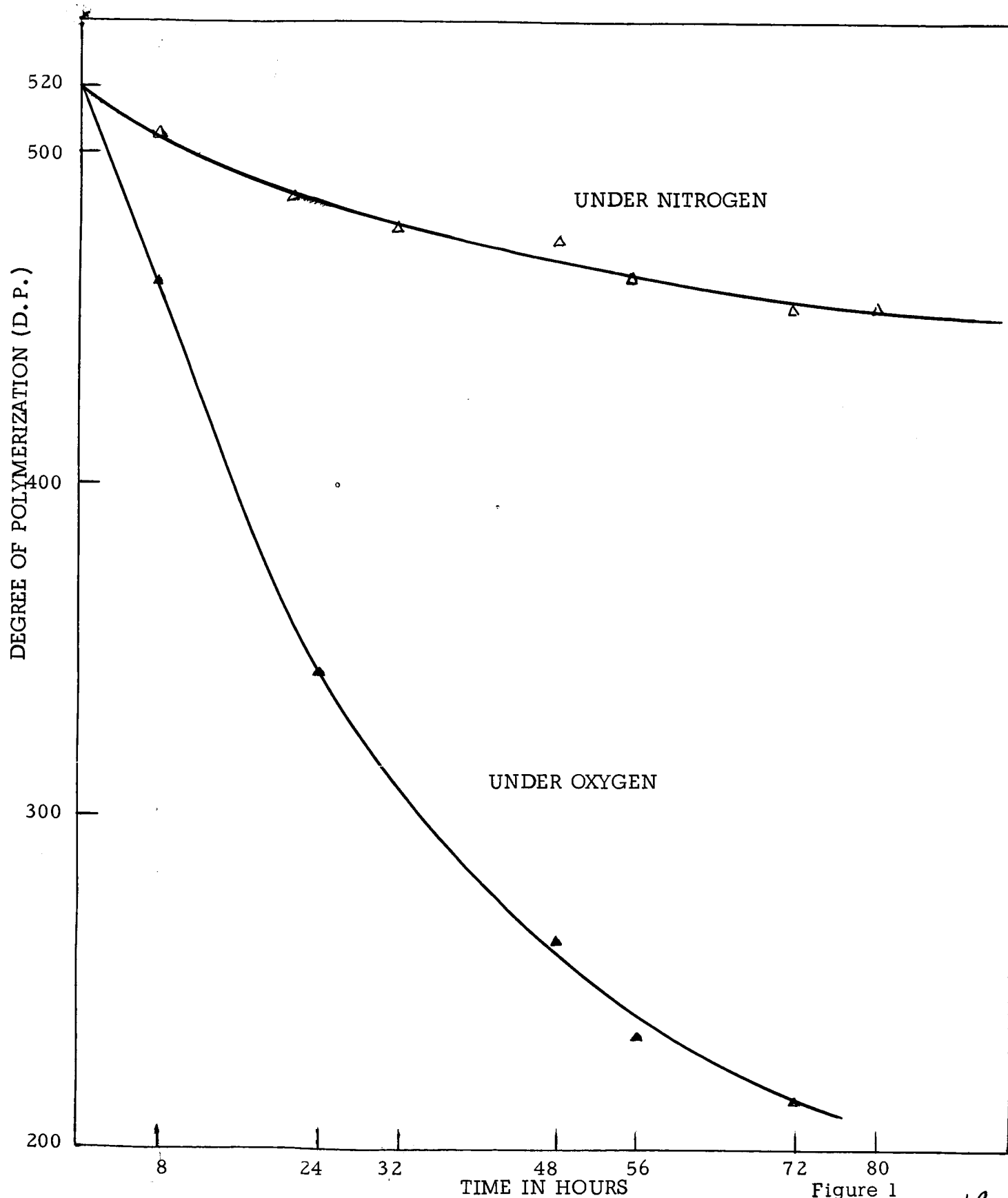
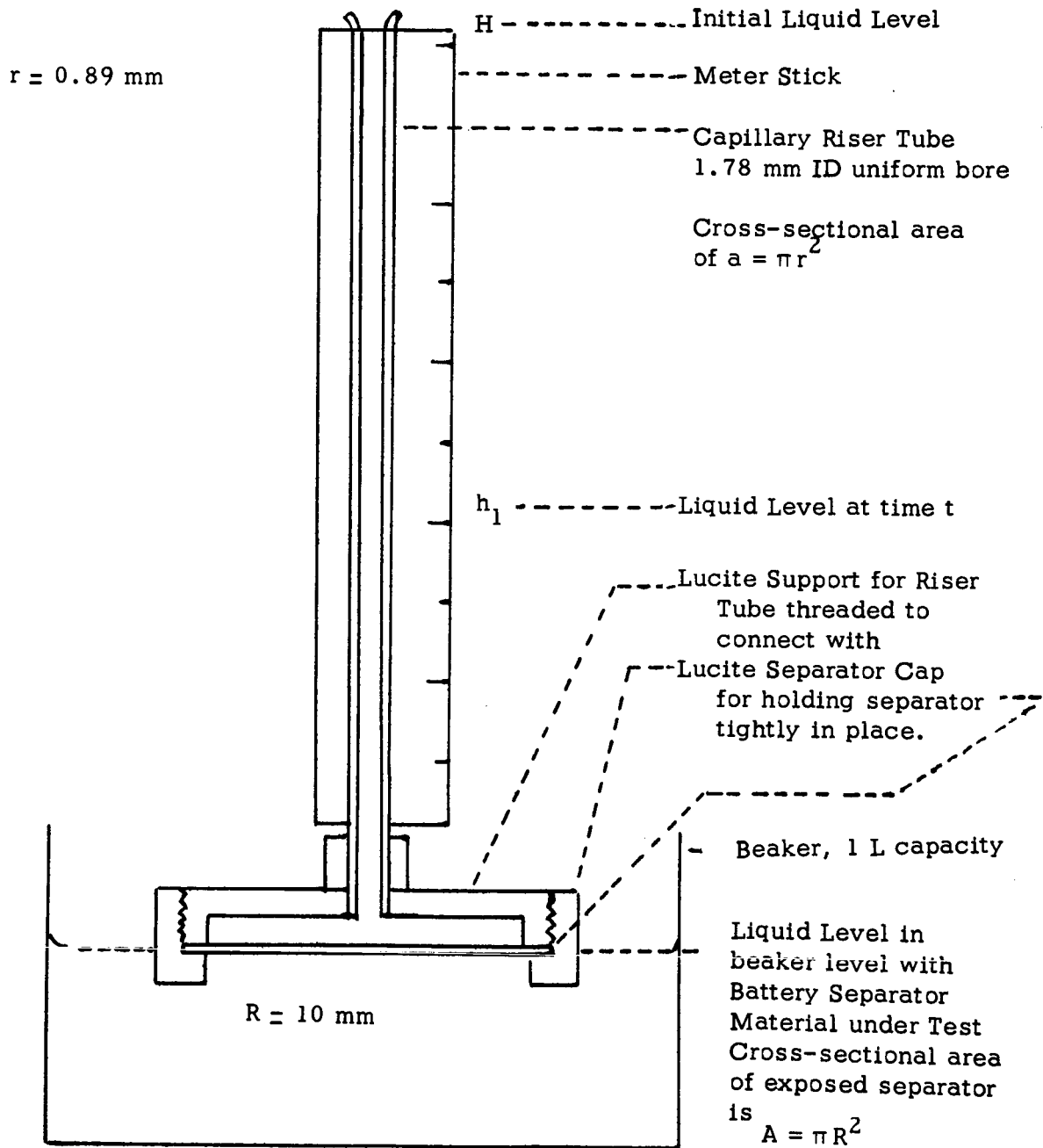


Figure 1



# PORE DIAMETER TEST ASSEMBLY

Figure 3



- Notes: 1. Volume of beaker must be large enough so that flow of liquid into beaker through separator does not alter the liquid level in the beaker significantly.
2. Air bubbles below separator must be displaced carefully.



# POROSITY DETERMINATION

DATE: 6-7-63 RI# 7054 AMP# 211 WT. SAMPLE 0.0500 G.  
 IDENTIFICATION: SYNFOR - (0.009" thick)  
 MICRON BREAK μ, TOTAL POROSITY 2.80 cc./g. % POROSITY      %

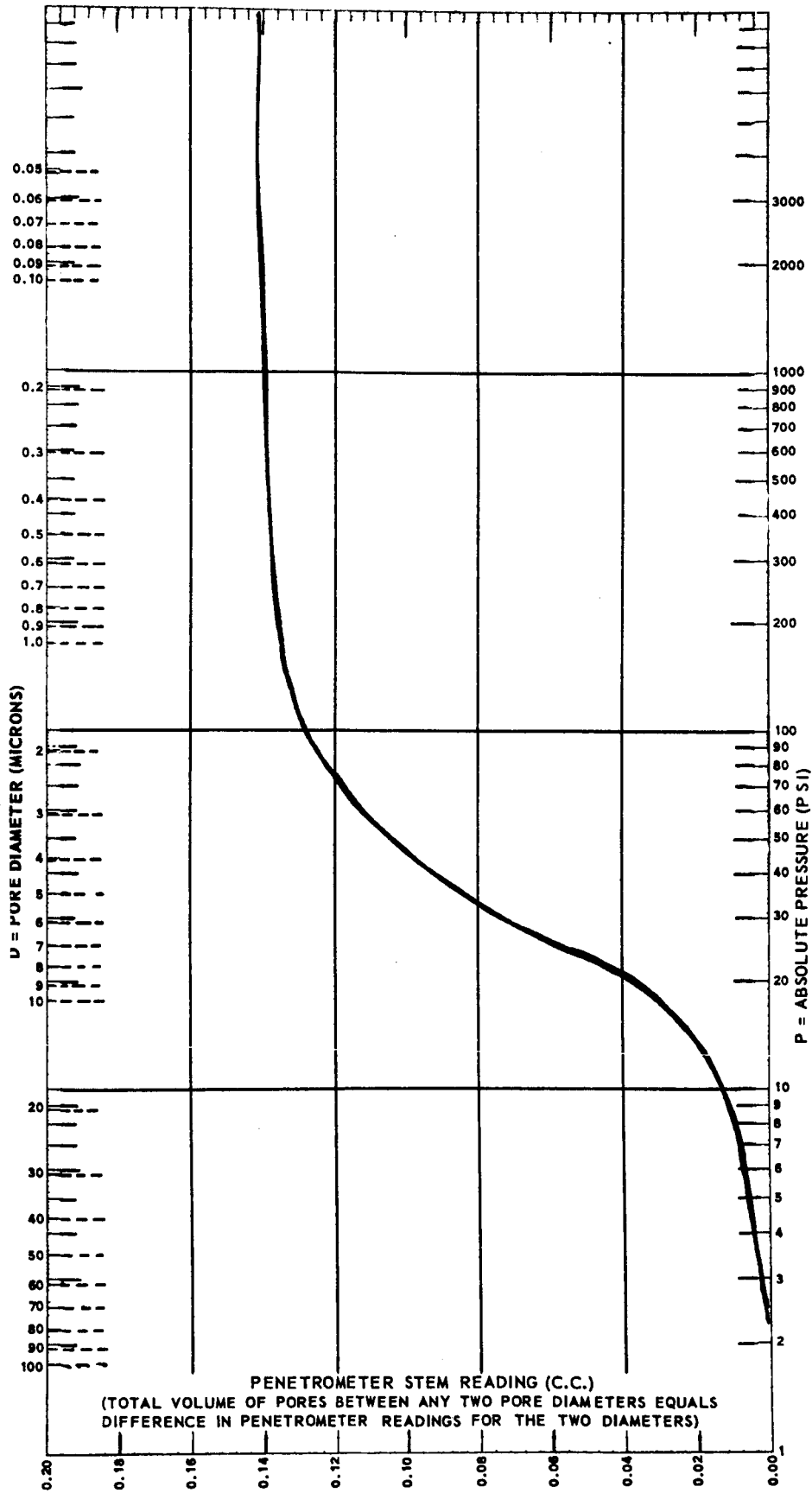


Figure 4

# POROSITY DETERMINATION

DATE: 6-28-63 RI# P.O. 7054 WT. SAMPLE 0.100 G.  
 IDENTIFICATION: ACROPOR GELMAN (WA)  
 MICRON BREAK 1.03 cc/g, % POROSITY      %

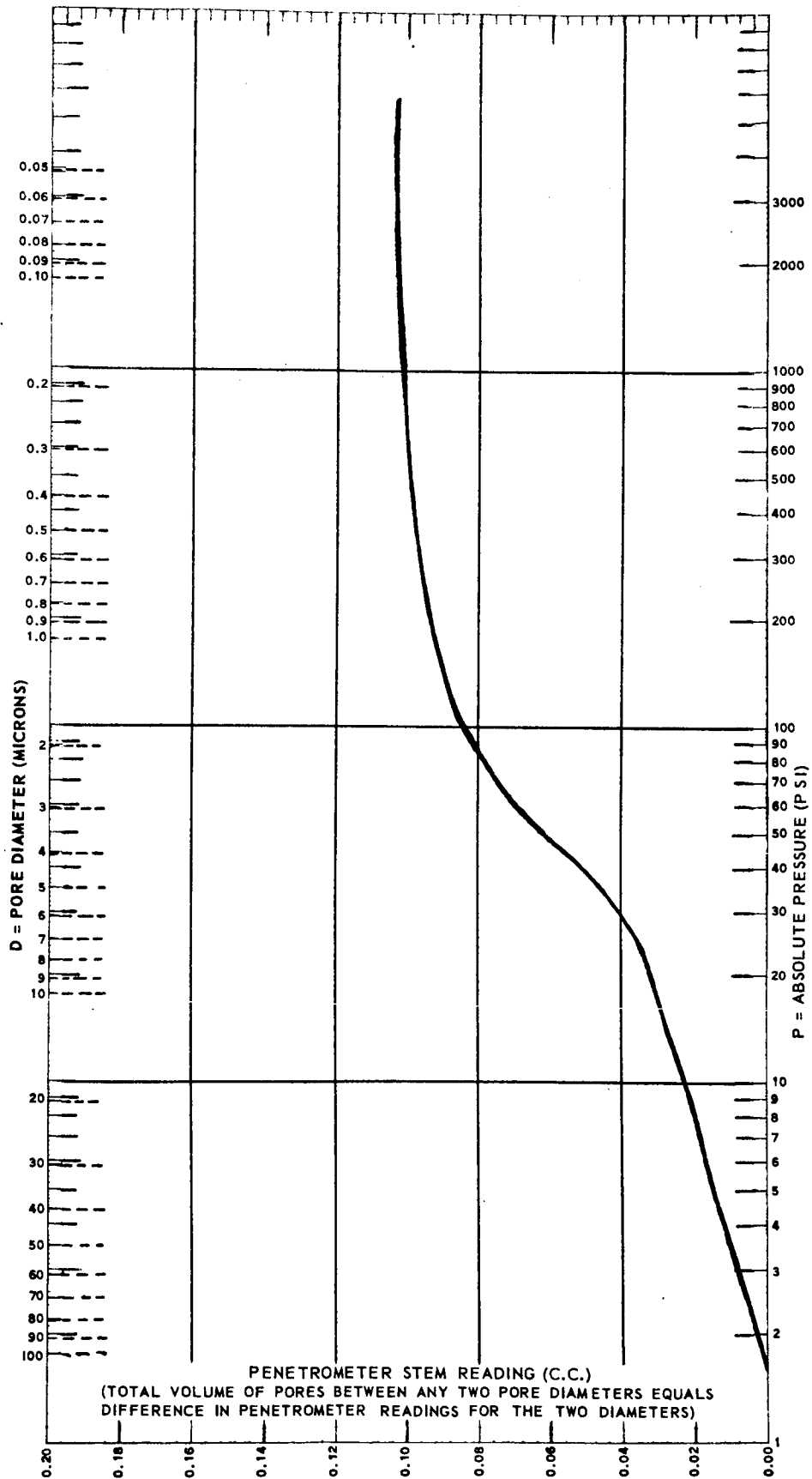


Figure 5

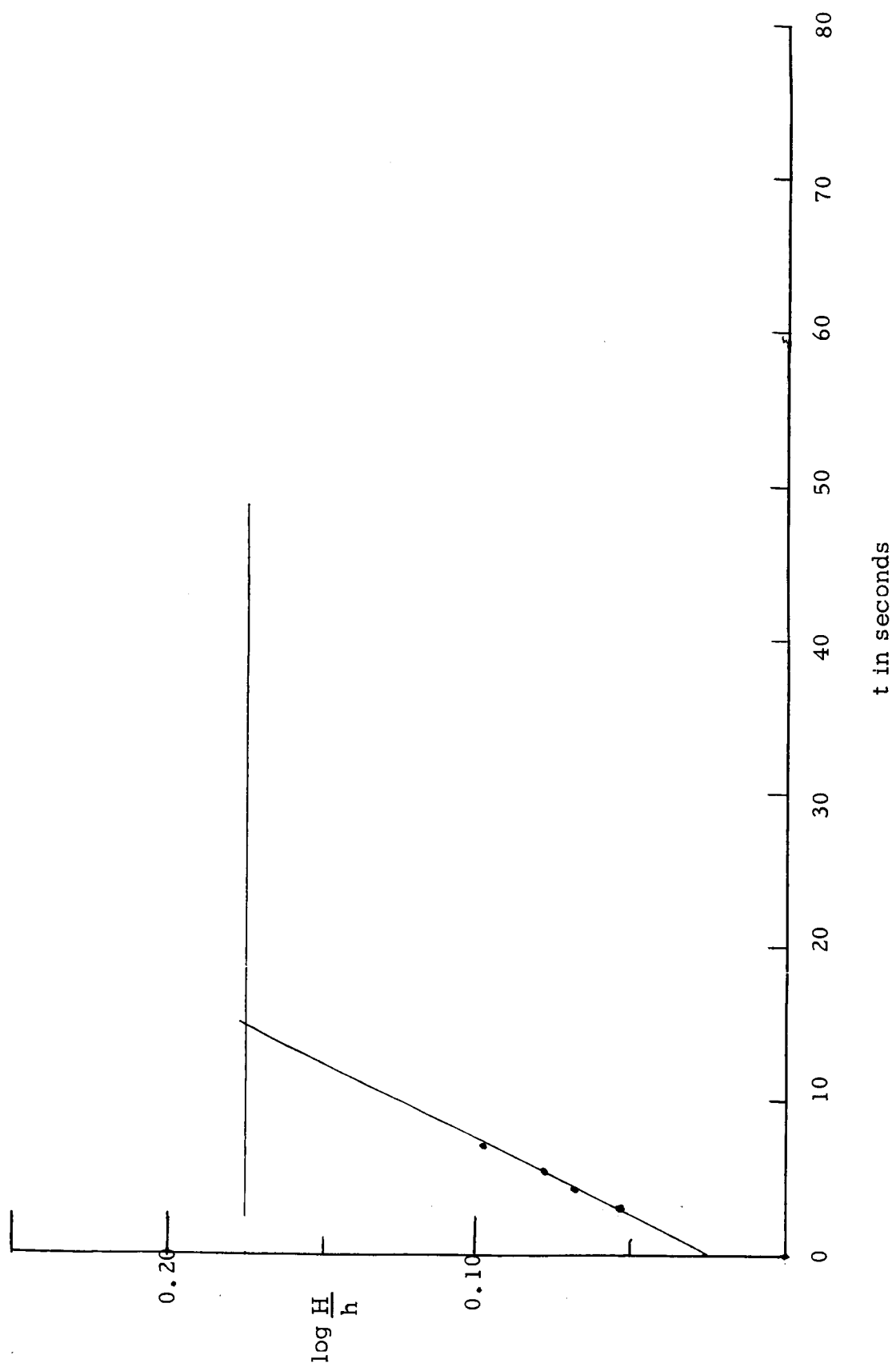
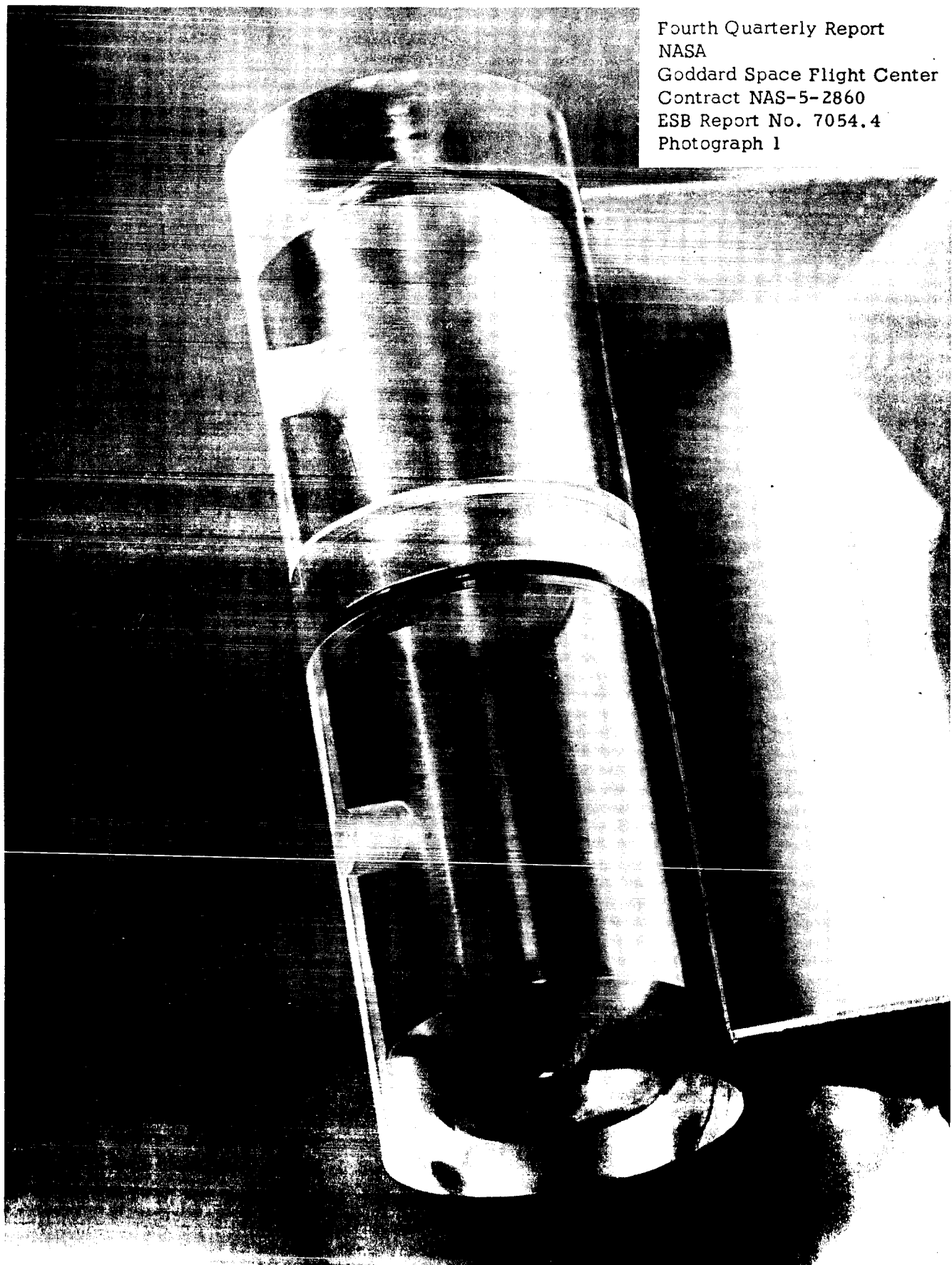


Figure 6

Fourth Quarterly Report  
NASA  
Goddard Space Flight Center  
Contract NAS-5-2860  
ESB Report No. 7054.4  
Photograph 1



Fourth Quarterly Report  
NASA  
Goddard Space Flight Center  
Contract NAS-5-2860  
ESB Report No. 7054.4  
Photograph 2

